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COMPARISON OF SAMPLER COLLECTION EFFICIENCY MEASUREMENTS USING A POLYDISPERSE SOLID AEROSOL AND A MONODISPERSE LIQUID AEROSOL

Jana Kesavan

INDEPENDENT CONTRACTOR

Robert W. Doherty

RESEARCH AND TECHNOLOGY DIRECTORATE

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Aberdeen Proving Ground, MD 21010-5424

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Kesavan,* Jana; and Doherty, Robert W.

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13. ABSTRACT *(Maximum 200 words)*

This study was conducted to determine the merits of using a single test of a polydisperse solid aerosol in place of multiple tests of monodisperse liquid aerosols in the characterization of aerosol sampling systems. The polydisperse aerosol chosen was an aluminum oxide (Al_2O_3) commercial abrasive. Analysis of the Al_2O_3 particles was by a Coulter Particle Size Analyzer. The monodisperse aerosols were fluorescent oleic acid droplets generated by a vibrating orifice aerosol generator (VOAG). Analysis of the fluorescent monodisperse particles was by UV fluorometry. The results show that over the 2-6 micron size range there is sufficiently good agreement between the rapid polydisperse solid aerosol method and the labor intensive liquid monodisperse aerosol method to warrant the use of the polydisperse test as a rapid screening device. Above 6 microns, the solid particles show evidence of bounce and re-entrainment into the aerosol flow. In turn, this results in a higher than expected sampling efficiency for solid particles for the particular aerosol sampler tested.

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PREFACE

The work described in this report was authorized under Project No. 622384/ACB2, Non-Medical CB Defense and Contract No. DAAD13-98-M-0083. This work was started in September 1998 and completed in May 2000.

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COMPARISON OF SAMPLER COLLECTION EFFICIENCY MEASUREMENTS USING A POLYDISPERSE SOLID AEROSOL AND A MONODISPERSE LIQUID AEROSOL

1. INTRODUCTION

Air sampling is conducted to determine the concentration of airborne material in the air. For example, air sampling is conducted to monitor the toxic material exposure to workers in occupational settings, to monitor possible chemical and/or biological agent exposure to soldiers in battlefields, and to monitor particulate matter exposure to the general population in both indoor and outdoor environments. Many kinds of air samplers are used to sample air. These samplers collect liquid and solid particles from air for further analysis or in some cases display the results immediately.

The performance of a sampler depends on the sampler's aspiration, transmission, and collection efficiencies. The aspiration efficiency of a sampler gives the efficiency with which particles enter into the sampler inlet; transmission efficiency gives the efficiency with which the particles are transported to the collection point, and the collection efficiency gives the efficiency with which particles are captured and retained by the sampling medium. The performance of a sampler is a product of aspiration, transmission, and collection efficiencies.

Many methods have been used by researchers to characterize samplers. Chen et al. (1985) characterized a virtual impactor using a mixture of monodisperse polystyrene latex particles (PSL) with the Aerodynamic Particle Sizer (APS) for analysis and fluorescent liquid dioctyl phthalate (DOP) with fluorometer analysis. John and Kreisberg (1999) also characterized samplers using aerosolized PSL particles and an APS for analysis. Maynard et al. (1999) used polydisperse glass microspheres and an APS for determining sampler penetration.

The APS can only be used in sampler characterization studies where the concentrated output of the sampler remains as an aerosol. Some samplers that sample for biological material collect the aerosol into a liquid and an APS could not be used in this situation. In addition, the losses in the connecting tube between the APS and the sampler have to be minimized for accurate measurements and the number of small particles has to be low enough to avoid 'phantom' particles due to coincidence counting.

Narrowly disperse Al_2O_3 (geometric standard deviation $\approx$ 1.35) aerosol and a gravimetric analysis were used by Aizenberg et al. (2000) to determine the sampling efficiency of personal inhalable aerosol samplers in wind tunnels. Buchan et al. (1986) determined the sampling efficiencies in a wind tunnel for open and closed face cassettes as well as an experimental cassette using solid particles and gravimetric analysis. Solid particles used in these tests were iron (mass median aerodynamic diameter (MMAD) = 2.4 μm and geometric standard deviation (GSD) = 1.68), tungsten (MMAD = 9.0 μm and GSD = 1.56), and aluminum spheres (MMAD = 24.0 μm and GSD = 1.61).

McFarland et al. (1987) used fluorescein tagged oleic acid aerosol and fluorometry to characterize samplers. In this study, McFarland et al. also used solid glass beads of 24.9 μm aerodynamic diameter with Coulter analysis method to determine penetration efficiency for solid particles. However, the solid and liquid particle sizes did not cover the same particle size range to allow a comparison between the effects of using solid vs. liquid particles.

Disadvantages of using monodisperse particles includes the expense in purchase of the monodisperse material, the equipment necessary to disseminate the particles, and the necessity of doing separate experiments for each particle size. These problems can be overcome by using a solid

polydisperse aerosol with a Coulter analysis. Glass beads, Al_2O_3, and polydisperse mixes of PSL are some of the polydisperse solid particles that can be used in sampler characterization.

A reference sample is required when the sampling efficiency is experimentally determined. A filter sample is taken as a reference sample along with the sampler under test. Full removal of particles from the filters is required for the analysis. We have conducted tests to validate the method of recovery of solid particles from polycarbonate membrane filters (Osmonics Inc., Minnitonka, MN) for a Coulter analyzer analysis (Kesavanathan and Doherty 1999) and sodium fluorescein from glass fiber type A/E filters (Pall Corporation, Ann Arbor, MI) for fluorometry (Kesavan and Doherty 2000a). These particle removal methods were used in this study.

The objectives of the study are to compare sampler characterization methods using solid polydisperse particles with Coulter analysis, and fluorescent monodisperse liquid particles with fluorometric analysis. Polydisperse Al_2O_3 particles were used as the solid particles and fluorescent oleic acid particles were used as the monodisperse liquid particles.

2. METHOD

2.1 Sampler

The sampler used in this test is a SCP 1021 high volume sampler (SCP Dynamics, Inc., Minneapolis, MN). It has two concentration stages. The first stage has multiple acceleration nozzles and receiver ports. The second stage has a single slot acceleration nozzle and a receiver port. The first virtual impactor stage concentrates particles larger than 2.5 μm from a flow rate of 1000 L/min into a flow rate of 100 L/min. The second virtual impactor stage further concentrates particles larger than 2.5 μm into a 20 L/min air flow rate. This particle enriched 20 L/min air stream is directed through a long stem to a liquid impinger/bubbler containing 40 mL of liquid which has been designed to retain particles. Because a Coulter analyzer is used to determine the size of the collected particles, a Coulter electrolyte solution was used as the collection liquid.

The sampler's specified flow rate is 1000 L/min, however, a flow rate of 1350 L/min was measured. A voltage controller was used to modify the power to the sampler to lower the actual flow rate of the sampler to the specified flow rate of 1000 L/min.

2.2 Polydisperse Solid Aerosol, Al_2O_3

Polydisperse irregularly shaped solid Al_2O_3 particles (Saint-Gobain Industrial Ceramics, Worcester, MA) were used in this study. Polydisperse Al_2O_3 particles were mixed to obtain approximately equal number of particles over the size range of 2 to 12 μm. A density of the 4 g/cm^3 was obtained for Al_2O_3 particles using an Autopycnometer (Micromeritics, Norcross, GA) (Kesavan and Doherty 2000b). From the literature, a dynamic shape factor correction of 1.22 was used for the Al_2O_3 particles (Vincent 1989). Both density and dynamic shape factor corrections were used to convert the volume equivalent diameter of the Coulter analyzer into aerodynamic diameter.

2.3 Coulter Multisizer™ II Analyzer

The Coulter Multisizer™ analyzer is a multichannel particle size analyzer that is used to measure solid particle size and number. It determines the number and size of particles suspended in a conductive liquid by monitoring the electrical current between two electrodes immersed in the conductive liquid on either side of a small aperture, through which a suspension of the particles is forced to flow. As each

particle passes through the aperture, it changes the impedance between the electrodes and produces an electrical pulse of short duration having a magnitude proportional to the particle volume. The series of pulses is electronically scaled, counted, and accumulated in a number of size related channels. The contents of the channels are displayed on the integral visual display in the form of a size distribution curve. The results can be selected to show particle volume, number, or surface area in either differential or cumulative form. The output of the multisizer can be sent to a computer for further analysis. A 50 μm diameter aperture that measures 1 – 30 μm diameter particles was installed in the system during the experiment.

Particles were collected in the electrolyte solution during sampling and the samples were stirred to obtain a uniform hydrosol concentration before each Coulter measurement. Each sample was measured three times. The Coulter analyzer was set to count the number of particles in 100 μL aliquots and the approximate time for counting particles in this volume is 10 seconds. Analyses with counting times greater than 10.5 seconds and less than 9.5 seconds were repeated. In most cases, longer counting times are due to partial or full blockage of the orifice. In addition, total raw count and total coincidence corrected count are displayed so that the operator can decide whether the sample has to be diluted.

Limitations on using a Coulter analyzer are that particles that are measured by the multisizer must not dissolve in the electrolyte solution, the orifice should not become partially or fully clogged during the measurement, the number concentration should be low enough so that there is no coincidence problems, and the density and the shape factor of the particles should be used to convert the volumetric size into an aerodynamic size.

2.4 Solid Aerosol Tests

Approximately 1.5 g of Al_2O_3 was aerosolized using a sonic nozzle in a 70 m^3 chamber. The aerosol was not neutralized in this study. SEM analyses of collected samples showed that the sonic nozzle de-agglomerates the particles into single particles (Figure 1).

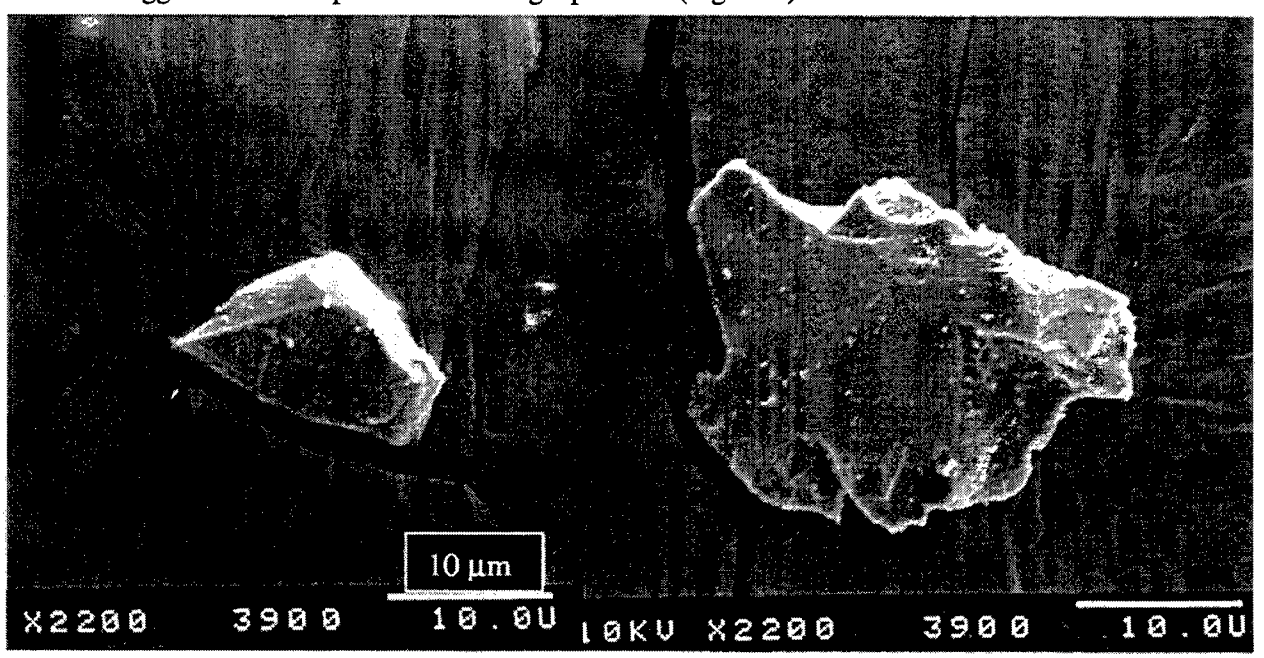

Figure 1. A SEM picture of typical Al_2O_3 particles. Only single particles were found, i.e., there were no agglomerated particles.

After the end of aerosol generation, the chamber air was mixed for 1 minute before sampling and also mixed for brief periods during the test to obtain uniform aerosol concentration using two mixing fans. Polycarbonate membrane filters were used as reference filters. The air flow rate of the reference filters were measured using an air flow meter (Buck calibrator, A.P. Buck, Inc., Orlando, FL).

A minimum of 7 minutes of sampling time was chosen because it takes some time to stabilize the flow rate of the sampler when turned on, and also takes some time to wind down when shut off. The sampler and the reference filter (Osmonics Inc., Minnitonka, MN) sampled the aerosol for the same amount of time.

Particle removal from the membrane filters was performed using the KD shaking method (Kesavanathan and Doherty 1999) which consists of 50 seconds of vortexing followed by 10 seconds of handshaking repeated for 5 minutes. Particle size and number concentration of the particles in liquid was determined using the Coulter analyzer. Although particles from earlier tests were undoubtedly present on the walls and nozzles of the SCP sampler, they were demonstrably shown not to be a source of interference between tests. This was shown by blank runs between tests.

Solid particles collected by the sampler into the liquid and particles removed from the reference filters into the liquid were analyzed using the Coulter analyzer. The Coulter analyzer gives the number and/or mass concentration as a function of volume equivalent particle diameter and was converted to an aerodynamic diameter using the following equation,

$$d_{aer} = d_{vol}\frac{\sqrt{\rho}}{SF}$$

d_{aer} – particle aerodynamic diameter
d_{vol} – volume equivalent diameter
ρ – density
SF – dynamic shape factor.

The sampling efficiency was determined by comparing the number of particles collected in the sampler liquid to the number of particles collected on the reference filter, normalized by the respective flow rates and liquid volumes.

2.5 Monodisperse Liquid Aerosol, Fluorescein Tagged Oleic Acid Aerosol

Monodisperse liquid fluorescent oleic acid aerosol was generated using the vibrating orifice aerosol generator (VOAG) (TSI Inc., St. Paul, MN) and the aerosol was delivered directly into the chamber for 10 minutes. An APS measured the particle size during the test, and an impactor sampled the particles onto a glass slide for microscopic evaluation. A microscopic picture of fluorescent oleic acid droplets on a slide is shown in Figure 2.

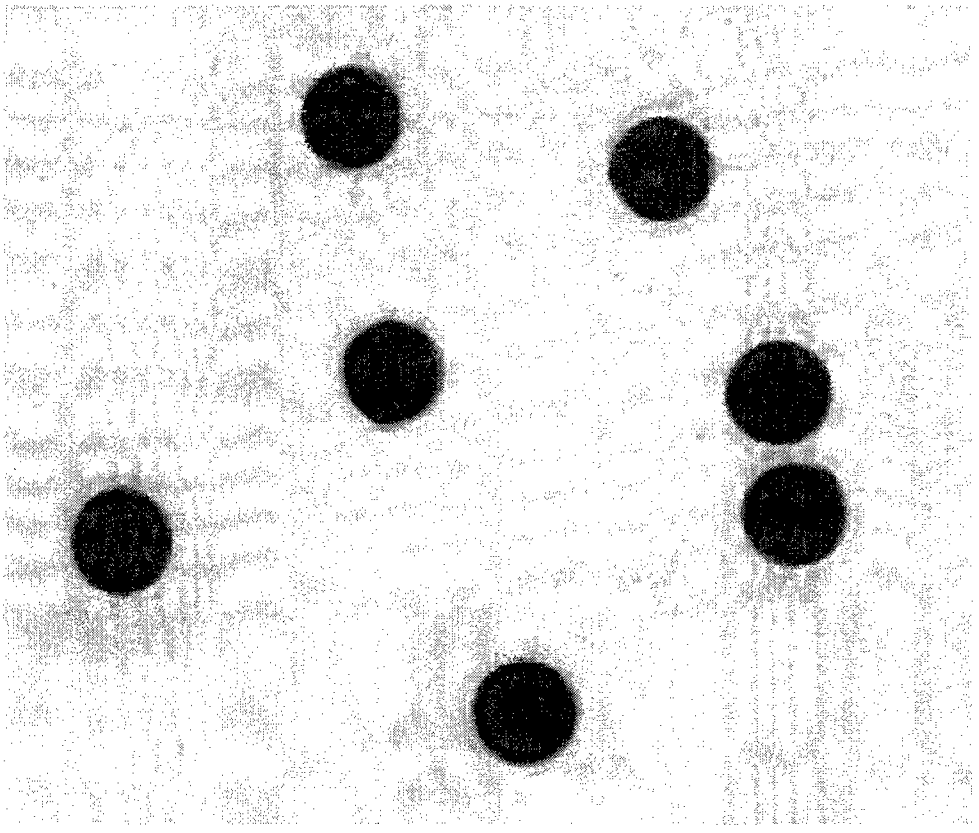

Figure 2. A microscopic picture of 10 μm aerodynamic diameter fluorescent oleic acid droplets.

The measured particle diameter was converted to aerodynamic particle size using a spread factor (Olan-Figueroa et al., 1982) and the density of fluorescent oleic acid.

The particle recovery solution that was used has equal amounts of alcohol and water and a pH between 8 and 10, obtained by adding a small amount of NH_4OH (e.g. 500 mL of 2-propanol + 500 mL of water + 0.5625 mL of 14.8 N NH_4OH). The recovery solution was used both to remove the fluorescein captured by the 47 mm diameter filters and to serve as the capture liquid in the sampler.

Glass fiber filters (Pall Corporation, Ann Arbor, MI) were used in the fluorescein tagged oleic acid tests as the reference filters. The air flow rate of the reference filters were measured using an air flow meter (Buck calibrator, A.P. Buck, Inc., Orlando, FL). The sampler and the reference filters sampled the air for the same amount of time, approximately 10 minutes. Reference filters were removed from the filter holders and were put into the recovery solution to remove the fluorescein from filters for fluorometry. The sampler collected the particles directly into 40 mL of recovery liquid. The liquid was removed and the fluorescence was measured using a fluorometer (Barnstead/Thermolyne, Dubuque, IA). This method is described by Kesavan and Doherty (2000a).

The sampling efficiency of each sampler was determined by comparing the sample collected by the sampler to that collected by the reference filter. The air flow rate and the liquid volumes were taken into account in the sampler efficiency calculations.

3. RESULTS

Figure 3 shows the sampling efficiency of the SCP1021 sampler using the polydisperse solid Al_2O_3 particles with Coulter analysis, and monodisperse fluorescein liquid particles with fluorometric analysis.

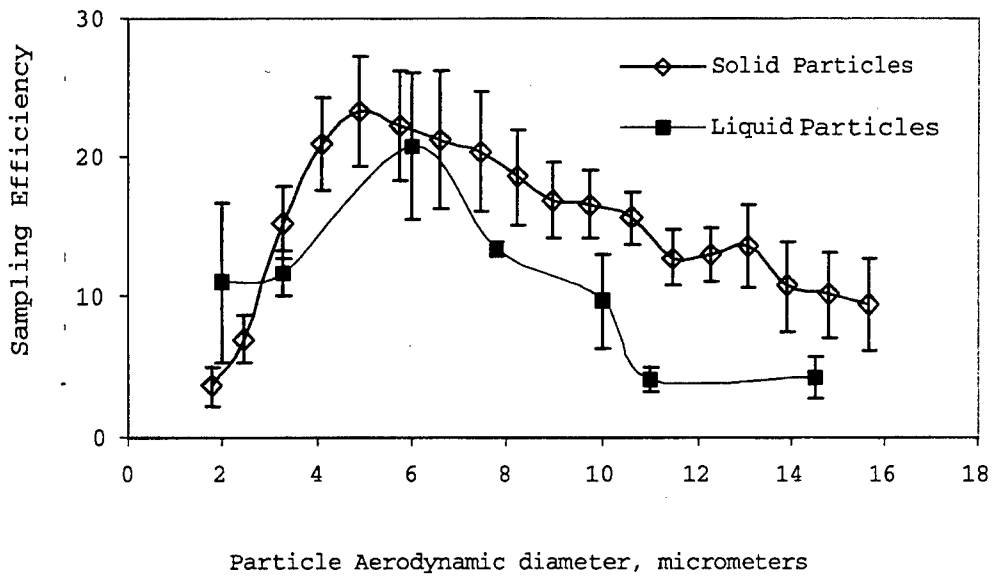

Figure 3. Sampling efficiency of sampler SCP 1021 measured using solid and liquid particles.

The sampling efficiency curves show that the sampling efficiency peaks at approximately 5-6 μm. In general, the sampling efficiency is lower for particles smaller than 6 μm because smaller particles are discharged with the secondary air flow of the SCP virtual impactors. In addition, the sampling efficiency of larger particles is low because of internal losses. This trend is seen for solid as well as for liquid particles. More specifically the results of Figure 3 also show that for particles smaller than 6 μm, there is little difference between using solid and liquid particles, however, for larger particles the sampling efficiency is higher for solid particles. This is most likely due to solid particle bounce and re-entrainment into the air flow.

4. DISCUSSION

Polydisperse Al_2O_3 particles were used as an aerosol in this study, however, any non-soluble polydisperse solid particle can be used as the test aerosol when a Coulter analyzer is used. One test using a polydisperse aerosol gives information on a range of particle size sampling efficiencies. However, solid particles may overestimate sampler efficiency at larger particle sizes. The use of monodisperse liquid particles provides a more conservative estimate of sampler efficiency, but requires much more time for testing each size separately.

Particles generated by the VOAG pass through a neutralizer before they enter the chamber to greatly reduce the charge on the particles. However, the Al_2O_3 particles were not electrically neutralized after aerosolization. The effect of charged particles on overall sampling efficiency of a sampler is

unknown though for particles greater than 6 microns, charge attractions should be minor compared to air drag and inertial forces. Future tests should neutralize Al_2O_3 particles before the particles enter the chamber to be used as a test aerosol.

By visual inspection, particle losses were present in the long stem that transported the aerosol to the liquid in the collection cup. Washing of this stem showed that similar amounts of fluorescein were recovered from the stem as from the collection liquid.

5. CONCLUSION

Reasonably good results can be rapidly obtained using a polydisperse solid Al_2O_3 aerosol, but consideration of the required accuracy of the results, especially at the larger particle sizes, must be taken into account before selecting either the polydisperse solid or monodisperse liquid aerosol approach.

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